PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of:

Applicants : David B. Smathers, Francis S. Valent, and Michael J. Regan
Serial No. : 10/527,513
Filed : October 26, 2005
Title : PROCESS FOR MAKING DENSE MIXED METAL Si_3N_4 TARGETS
Docket : 020324 223P2
Examiner : Jie Yang
Art Unit : 1793
Customer No.: 33,805

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

Sir/Madam:

Declaration Under 37 CFR §1.131

4624 Acwell Loop Grove City 43123

I, Francis S. Valent, of ~~6023 Epernay Way~~, ~~Galloway~~, Ohio ~~43119~~, am a citizen of the United States. I am a co-inventor of the above identified patent application. I hereby agree to and concur with the statements made by David B. Smathers in his Declaration Under 37 CFR §1.131 and attached Exhibits A and B thereof.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signature: Francis S. Valent Date: 11/14/2008
Francis S. Valent

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P.O. Box 1450
Alexandria, Virginia 22313-1450

Sir/Madam:

Declaration Under 37 CFR §1.131

I, David B. Smathers, do declare and state as follows:

[0001] I received a Bachelor of Science Degree in Physics from Rhodes College, a Masters of Science Degree in Materials Science from the University of Wisconsin at Madison, and received my Doctor of Philosophy Degree in Materials Science from the University of Wisconsin, Madison.

[0002] I am presently the Director of Quality and Engineering at Tosoh SMD, Inc., one of the co-owners of the above patent application.

[0003] I am one of the named co-inventors of the above-identified patent application.

[0004] The inventors, including me, conceived and reduced to practice in the United States, the invention claimed in the above-identified patent application, prior to June 17, 2002.

[0005] Exhibit A attached is a copy of an invention disclosure that I executed along with one of the other co-inventors prior to June 17, 2002. This exhibit is evidence of both conception and actual reduction to practice of the claimed invention. Certain portions of this document have been redacted to remove non-relevant confidential subject matter and to conceal specific dates. This exhibit refers to a process for making sputtering targets wherein the claimed metal powder

and Si_3N_4 powder were blended with MgO or SiO sintering aids and that this blend was then pressure consolidated under heated conditions to form a blank of greater than 95% actual density.

[0006] One specific embodiment of this invention referred to on page 2 of the exhibit under portion "D" thereof refers to a tungsten, silicon nitride, magnesium oxide blend that was screened, vacuum hot pressed and ground into a sputter target blank. The blank was then solder bonded to a Cu/Cr backing plate using indium solder. A copy of the blueprint for this target is attached as Exhibit B with the date redacted. Accordingly, based on this information, the subject matter of the above identified application as claimed therein was conceived and reduced to practice prior to June 17, 2002.

[0007] I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signature: _____ Date: June 23, 2008

David B. Smathers

TOSOH SMD, INC.----------DISCLOSURE OF INVENTION

This is an important legal document. See opposite side for definitions of asterisked terms. The form should be carefully completed by the inventor(s) and forwarded in duplicate to the Patent Representatives

1. DESCRIPTIVE TITLE OF INVENTION

Process for Making Dense Mixed Metal-Si3N4 Targets

2. NAMES(S), TITLE(S) & HOME ADDRESS(ES) OF INVENTOR(S) include full middle name, COUNTRY of citizenship, and badge number.
David Bird Smathers, 3298 Kirkham Road, Columbus, OH 43123; U.S.A. Citizen; TSMD Badge 86459
Francis Samuel Valent, 6023 Epernay Way, Galloway, OH 43119; U.S.A. Citizen; TSMD Badge 86394

3. EMPLOYER: Tosoh SMD, Inc. 3600 Gantz Road, Grove City, Ohio 43123 USA Phone 614-875-7912

4. STAGE OF DEVELOPMENT	DATE (Month/Year)	LOCATION	IDENTIFY PERSONS OR RECORDS SUPPORTING FACTS IN 4.A-E
A. First disclosure to others		TSMD/HP	Proposal to Hewlett Packard under CDA.
B. First sketch or drawing		TSMD/HP	Quotation
C. First written description		TSMD	Quotation/Order with Part Routing and B.O.M.
D. Completion of first model or device		TSMD /HP	Lot 9H0061-101 3" Sample
E. First actual reduction to practice		TSMD /HP	Lot 9H0101-101 RMX-12 Version

5. LIST PROJECT NUMBER AND OTHER PERTINENT NOTEBOOK ENTRIES, PHOTOGRAPHS, REPORTS, DRAWINGS:
NPD 780-1;804-1; 828-1; 871-1; 871-2; 871-4; 1042-1 All contain MgO
NPD 736-1; 737-1; 818-1; 858-1; 858-2 Contain no MgO or uses an Alternative (SiO)
Spreadsheet containing article attributes – Shared with HP and all versions are password protected.
Density Evaluation and Correction Document
Report on the evaluation of a used X-901 target to HP .
HP Drafted a Specification in . but it has never been issued and is currently being re-written by Garold Radke to cover current design.
TXRD Analysis of Targets for phase identification

6. IF THE INVENTION WAS DISCLOSED OUTSIDE OF TOSOH, identify the individuals, the companies or activities they represent, and the date of disclosure
Mike Regan, Hewlett Packard Corvallis, Advanced Research Lab, Lead Engineer
Jim Roberts, Hewlett Packard Corvallis, Advanced Research Lab, Procurement Specialist
Garold Radke, Hewlett Packard Corvallis, Advanced Research Lab, Lead Engineer
Bob Strain
Eldon Hilton
Judy Thompson
Marzio LeBan

7. LIST ANY KNOWN PUBLIC USE, PUBLICATION, OR ORAL PRESENTATION OF THE INVENTION, SALE OR OFFER FOR SALE

	No	Yes	Date	Persons, Companies, or Publications
A. Sold	X			Proprietary sale under CDA
B. Offered for sale as part of a product*	X			
C. Offered for sale in development program*		X		Several parts have been sold to HP to cover manufacturing expenses as HP works to determine composition and platform best suited to their process.
D. Described in a publication*	X			
E. Submitted to a publication*	X			
F. Placed in a public use*	X			
G. Used to make a product in public use	X			
H. Orally presented	X			

8. LIST DATES & DETAILS OF ACTIVITIES OF 7.A-H IF SCHEDULED IN THE FUTURE

9. RELATED GOVERNMENT CONTRACT(S) Did your job assignments involve work under a government contract related to the inventive subject matter at the time the invention was . . .
Conceived ? Yes____ NoX____ Contract Number_____
First successfully tested ? Yes No Contract Number

10. ATTACH A CONCISE TECHNICAL DESCRIPTION OF THE INVENTION. THE DESCRIPTION SHOULD INCLUDE:
A. General purpose of the invention
B. Prior art (previous) methods, materials, or devices performing function of the invention
C. Disadvantages of prior art
D. Identification of component parts, or steps, and explanation of mode of operation of invention
E. Alternate embodiments of the invention
F. Advantages of the invention over prior art
G. Features of the invention believed to be new
H. If a joint invention, the contribution of each inventor
The completed description should be signed by the inventor(s) and then read and signed by a technically competent witness, using the statement *DISCLOSED TO AND UNDERSTOOD BY ME THIS ___DAY___OF___19___." Drawings, sketches, photographs, reports, if available, may form a part of the disclosure, and reference thereto can be made to complete this description.

11. RECOMMENDED SECURITY CLASSIFICATION OF THE INVENTION
Unclassified Confidential Secret Other specify

12. SIGNATURE(S) OF INVENTOR(S) AND DATE

David B Smathers

Francis S. Valent

SIGNATURE(S) OF WITNESS(ES) AND DATE

J E Murphy

EXHIBIT
tabbies
A

Concise Technical Description of Invention

Descriptive Title: Process for Making Dense Mixed Metal-Si_3N_4 Targets

Inventor(s): David B. Smathers; Frank Valent

A. General Purpose of the Invention
As part of a joint development program with Hewlett Packard Ink Jet Business Unit Advanced Research Lab, TSMD developed metal/ceramic targets for the heater layer in ink jet printer heads. HP wanted mixtures of Ta-Al-O, Ta-Si-N or W-Si-N. The targets were required to be more than 95% dense and in a variety of target platform geometries.

The basic invention is a near full density Tungsten or Tantalum mixture with Silicon Nitride using the Vacuum Hot Press and a sintering aid. The choice for the sintering aid that works best is Magnesium Oxide between 0.05 to 4 weight percent with respect to the Silicon Nitride content.

B. Prior Art (previous) Methods, Materials, or Devices Performing Function on this Invention
The targets have been fabricated using VHP or HIP with low density results. Praxair MRC filed a patent on a HIP method using a pre-densification method. This did not work; the target was not dense and cracked during sputtering. According to Mark Gore, Unaxis also worked on this material using HIP without success.

C. Disadvantages of Prior Art
Without the sintering aid, the Silicon Nitride will not densify. The target material is not strong enough to support the bonding and sputtering operations.
At the temperatures required for densification of the ceramic, normal container materials can not be used. The blend can be compacted and covered with a glass prior to HIP. After HIP, the glass has to be machined off. The material efficiency is not as good as in the VHP since there is no external container to constrain the part.

D. Identification of Component Parts, or Steps, and Explanation of Mode of Operation of Invention
The target consists of a mixture of between 50 and 70 at% Tungsten and 50 to 30 at% Silicon Nitride. Magnesium Oxide is added to the Silicon Nitride at rate of 0.05 to 4 weight per cent with respect to the Silicon Nitride weight.
The Si_3N_4 is first mixed with the MgO. The mixture is –325 Mesh.
The Si_3N_4/MgO mixture is blended with the Tungsten powder. The Tungsten powder is –100 mesh but is made up of individual powder particles nominally 1 to 5 microns in size.
The mixture is screened through a –50 mesh screen multiple times (at least twice) to minimize the size of agglomerated Si3N4 to less than 300 microns in diameter. The MgO is hydroscopic and causes the mixture to absorb water and clump up.
The mixture is formed into a target blank using the Vacuum Hot Press according to recipe 16. The press operates at 800 torr/1640C during the peak of the cycle. The 800torr Argon backfill is required to keep the nitrogen from decomposing out of the Si_3N_4.
The target blank is ground to thickness and ground to diameter.
The target blank is solder bonded to a Cu1Cr backing plate using Indium solder.

E. Alternate Embodiments of the Invention

SiO may be used in large amounts to densify the target. More than 6 at% SiO is required to get the target density higher than 85%.

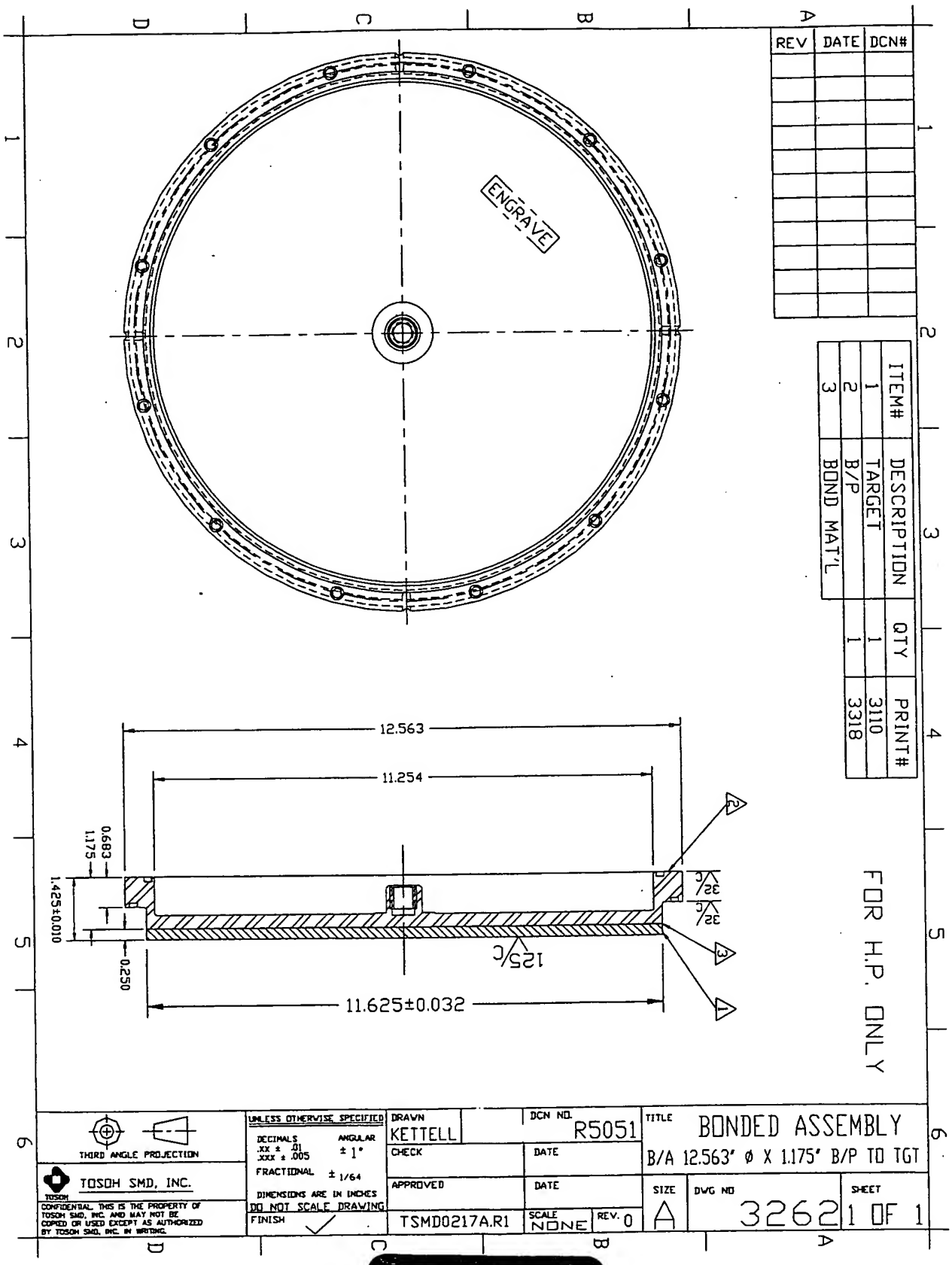
We could screen the powder under a protective atmosphere of nitrogen to limit the moisture pickup, use a variety of types of screening equipment designed to break up agglomerations such as a Sweeco vibrating head or use other powder blending techniques such as mechanical alloying to get better mixing of the different density components.

F. Advantages of the Invention Over the Prior Art

The MgO causes the Si_3N_4 to densify and the target stays intact during sputtering.
The VHP near net shape part improves material utilization.
The operation of the VHP at a slight over-pressure (800 torr) keeps the Nitrogen from escaping the mixture during the high temperature press cycle.

G. Features of the Invention Believed to be New

Inclusion of the MgO in the Si_3N_4/W mixture to form a dense sputtering target. The MgO does not harm the film in the application.
Screening of the mixture to control the Si_3N_4 agglomerates.
Operation of the metal mixture pressing under a protective atmosphere.



REV	DATE	DCN#

ITEM#	DESCRIPTION	QTY	PRINT#
1	TARGET	1	3110
2	B/P	1	3318
3	BOND MAT'L		

FOR H.P. ONLY

THIRD ANGLE PROJECTION

TOSOH SMD, INC.

UNLESS OTHERWISE SPECIFIED
DECIMALS .XX ± .01 .XXX ± .005
ANGULAR ± 1°
FRACTIONAL ± 1/64
DIMENSIONS ARE IN INCHES
DO NOT SCALE DRAWING
FINISH ✓

DRAWN	KETTELL	DCN NO.	R5051
CHECK		DATE	
APPROVED		DATE	
	TSMD0217A.R1	SCALE NONE	REV. 0

TITLE: BONDED ASSEMBLY
B/A 12.563" Ø X 1.175" B/P TO TGT

SIZE	DWG NO	SHEET
A	3262	1 OF 1

EXHIBIT
tabbies

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Art Unit : 1793
Customer No.: 33,805

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

Sir/Madam:

Declaration Under 37 CFR §1.131

I, Michael J. Regan, of 3210 NW Arrowhead Circle, Corvallis, Oregon 97330, am a citizen of the United States. I am a co-inventor of the above identified patent application. I hereby agree to and concur with the statements made by David B. Smathers in his Declaration Under 37 CFR §1.131 and attached Exhibits A and B thereof.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signature: Michael J. Regan
Michael J. Regan

Date: November 14, 2008

PATENT

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I, David B. Smathers, do declare and state as follows:

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[0002] I am presently the Director of Quality and Engineering at Tosoh SMD, Inc., one of the co-owners of the above patent application.

[0003] I am one of the named co-inventors of the above-identified patent application.

[0004] The inventors, including me, conceived and reduced to practice in the United States, the invention claimed in the above-identified patent application, prior to June 17, 2002.

[0005] Exhibit A attached is a copy of an invention disclosure that I executed along with one of the other co-inventors prior to June 17, 2002. This exhibit is evidence of both conception and actual reduction to practice of the claimed invention. Certain portions of this document have been redacted to remove non-relevant confidential subject matter and to conceal specific dates. This exhibit refers to a process for making sputtering targets wherein the claimed metal powder

and Si_3N_4 powder were blended with MgO or SiO sintering aids and that this blend was then pressure consolidated under heated conditions to form a blank of greater than 95% actual density.

[0006] One specific embodiment of this invention referred to on page 2 of the exhibit under portion "D" thereof refers to a tungsten, silicon nitride, magnesium oxide blend that was screened, vacuum hot pressed and ground into a sputter target blank. The blank was then solder bonded to a Cu/Cr backing plate using indium solder. A copy of the blueprint for this target is attached as Exhibit B with the date redacted. Accordingly, based on this information, the subject matter of the above identified application as claimed therein was conceived and reduced to practice prior to June 17, 2002.

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Signature: _____ Date: June 23, 2008

David B. Smathers

TOSOH SMD, INC.----------DISCLOSURE OF INVENTION

This is an important legal document. See opposite side for definitions of asterisked terms. The form should be carefully completed by the inventor(s) and forwarded in duplicate to the Patent Representatives

1. DESCRIPTIVE TITLE OF INVENTION

Process for Making Dense Mixed Metal-Si3N4 Targets

2. NAMES(S), TITLE(S) & HOME ADDRESS(ES) OF INVENTOR(S) include full middle name, COUNTRY of citizenship, and badge number.
David Bird Smathers, 3298 Kirkham Road, Columbus, OH 43123; U.S.A. Citizen; TSMD Badge 86459
Francis Samuel Valent, 6023 Epernay Way, Galloway, OH 43119; U.S.A. Citizen; TSMD Badge 86394

3. EMPLOYER: Tosoh SMD, Inc. 3600 Gantz Road, Grove City, Ohio 43123 USA Phone 614-875-7912

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NPD 736-1; 737-1; 818-1; 858-1; 858-2 Contain no MgO or uses an Alternative (SiO)
Spreadsheet containing article attributes – Shared with HP and all versions are password protected.
Density Evaluation and Correction Document
Report on the evaluation of a used X-901 target to HP .
HP Drafted a Specification in . but it has never been issued and is currently being re-written by Garold Radke to cover current design.
TXRD Analysis of Targets for phase identification

6. IF THE INVENTION WAS DISCLOSED OUTSIDE OF TOSOH, identify the individuals, the companies or activities they represent, and the date of disclosure
Mike Regan, Hewlett Packard Corvallis, Advanced Research Lab, Lead Engineer
Jim Roberts, Hewlett Packard Corvallis, Advanced Research Lab, Procurement Specialist
Garold Radke, Hewlett Packard Corvallis, Advanced Research Lab, Lead Engineer
Bob Strain
Eldon Hilton
Judy Thompson
Marzio LeBan

7. LIST ANY KNOWN PUBLIC USE, PUBLICATION, OR ORAL PRESENTATION OF THE INVENTION, SALE OR OFFER FOR SALE

	No	Yes	Date	Persons, Companies, or Publications
A. Sold	X			Proprietary sale under CDA
B. Offered for sale as part of a product*	X			
C. Offered for sale in development program*		X		Several parts have been sold to HP to cover manufacturing expenses as HP works to determine composition and platform best suited to their process.
D. Described in a publication*	X			
E. Submitted to a publication*	X			
F. Placed in a public use*	X			
G. Used to make a product in public use	X			
H. Orally presented	X			

8. LIST DATES & DETAILS OF ACTIVITIES OF 7.A-H IF SCHEDULED IN THE FUTURE

9. RELATED GOVERNMENT CONTRACT(S) Did your job assignments involve work under a government contract related to the inventive subject matter at the time the invention was . . .
Conceived ? Yes____ NoX____ Contract Number_____
First successfully tested ? Yes____ No____ Contract Number_____

10. ATTACH A CONCISE TECHNICAL DESCRIPTION OF THE INVENTION. THE DESCRIPTION SHOULD INCLUDE:
A. General purpose of the invention
B. Prior art (previous) methods, materials, or devices performing function of the invention
C. Disadvantages of prior art
D. Identification of component parts, or steps, and explanation of mode of operation of invention
E. Alternate embodiments of the invention
F. Advantages of the invention over prior art
G. Features of the invention believed to be new
H. If a joint invention, the contribution of each inventor
The completed description should be signed by the inventor(s) and then read and signed by a technically competent witness, using the statement *DISCLOSED TO AND UNDERSTOOD BY ME THIS ___DAY___OF___19___." Drawings, sketches, photographs, reports, if available, may form a part of the disclosure, and reference thereto can be made to complete this description.

11. RECOMMENDED SECURITY CLASSIFICATION OF THE INVENTION
Unclassified Confidential Secret Other specify

12. SIGNATURE(S) OF INVENTOR(S) AND DATE

David B Smathers

Francis S. Valent

SIGNATURE(S) OF WITNESS(ES) AND DATE

J E Murphy

EXHIBIT
tabbies
A

Concise Technical Description of Invention

Descriptive Title: Process for Making Dense Mixed Metal-Si_3N_4 Targets

Inventor(s): David B. Smathers; Frank Valent

A. General Purpose of the Invention
As part of a joint development program with Hewlett Packard Ink Jet Business Unit Advanced Research Lab, TSMD developed metal/ceramic targets for the heater layer in ink jet printer heads. HP wanted mixtures of Ta-Al-O, Ta-Si-N or W-Si-N. The targets were required to be more than 95% dense and in a variety of target platform geometries.

The basic invention is a near full density Tungsten or Tantalum mixture with Silicon Nitride using the Vacuum Hot Press and a sintering aid. The choice for the sintering aid that works best is Magnesium Oxide between 0.05 to 4 weight percent with respect to the Silicon Nitride content.

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C. Disadvantages of Prior Art
Without the sintering aid, the Silicon Nitride will not densify. The target material is not strong enough to support the bonding and sputtering operations.
At the temperatures required for densification of the ceramic, normal container materials can not be used. The blend can be compacted and covered with a glass prior to HIP. After HIP, the glass has to be machined off. The material efficiency is not as good as in the VHP since there is no external container to constrain the part.

D. Identification of Component Parts, or Steps, and Explanation of Mode of Operation of Invention
The target consists of a mixture of between 50 and 70 at% Tungsten and 50 to 30 at% Silicon Nitride. Magnesium Oxide is added to the Silicon Nitride at rate of 0.05 to 4 weight per cent with respect to the Silicon Nitride weight.
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The mixture is formed into a target blank using the Vacuum Hot Press according to recipe 16. The press operates at 800 torr/1640C during the peak of the cycle. The 800torr Argon backfill is required to keep the nitrogen from decomposing out of the Si_3N_4.
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SiO may be used in large amounts to densify the target. More than 6 at% SiO is required to get the target density higher than 85%.

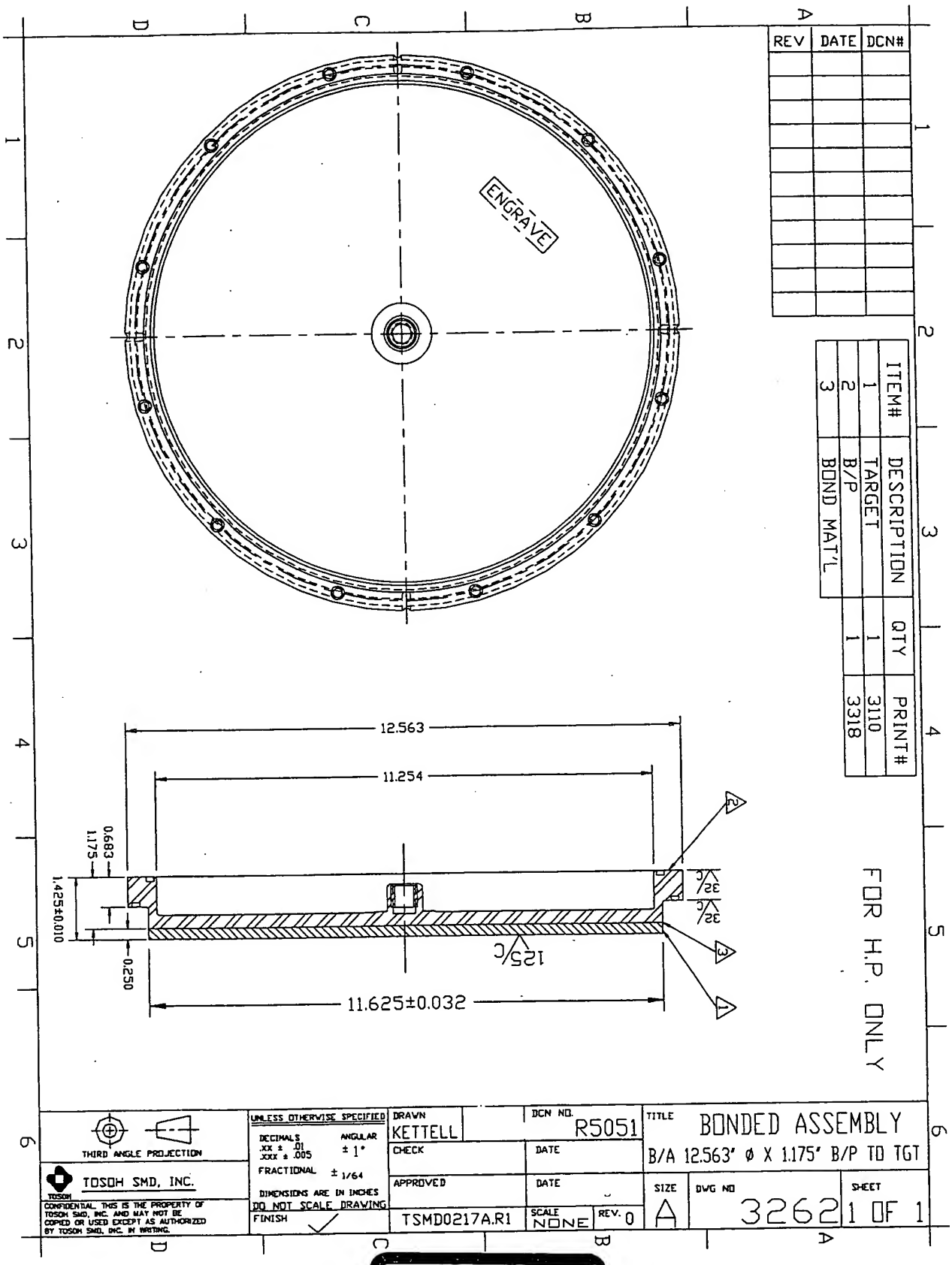
We could screen the powder under a protective atmosphere of nitrogen to limit the moisture pickup, use a variety of types of screening equipment designed to break up agglomerations such as a Sweeco vibrating head or use other powder blending techniques such as mechanical alloying to get better mixing of the different density components.

F. Advantages of the Invention Over the Prior Art

The MgO causes the Si_3N_4 to densify and the target stays intact during sputtering.
The VHP near net shape part improves material utilization.
The operation of the VHP at a slight over-pressure (800 torr) keeps the Nitrogen from escaping the mixture during the high temperature press cycle.

G. Features of the Invention Believed to be New

Inclusion of the MgO in the Si_3N_4/W mixture to form a dense sputtering target. The MgO does not harm the film in the application.
Screening of the mixture to control the Si_3N_4 agglomerates.
Operation of the metal mixture pressing under a protective atmosphere.



REV	DATE	DCN#

ITEM#	DESCRIPTION	QTY	PRINT#
1	TARGET	1	3110
2	B/P	1	3318
3	BOND MAT'L		

FOR H.P. ONLY

THIRD ANGLE PROJECTION

TOSOH SMD, INC.

CONFIDENTIAL. THIS IS THE PROPERTY OF TOSOH SMD, INC. AND MAY NOT BE COPIED OR USED EXCEPT AS AUTHORIZED BY TOSOH SMD, INC. IN WRITING.

UNLESS OTHERWISE SPECIFIED
DECIMALS .01 .XX ± .005 .XXX ± .005
ANGULAR ± 1°
FRACTIONAL ± 1/64
DIMENSIONS ARE IN INCHES
DO NOT SCALE DRAWING
FINISH ✓

DRAWN KETTELL		DCN NO. R5051	
CHECK		DATE	
APPROVED		DATE	
TSMD0217A.R1	SCALE NONE	REV. 0	

TITLE BONDED ASSEMBLY
B/A 12.563" ø X 1.175" B/P TO TGT

SIZE A | DWG NO 3262 | SHEET 1 OF 1

EXHIBIT
tabbies